

(11) (21) (C) **2,038,754**

(22) 1991/03/21

(43) 1991/11/05

(45) 1998/05/19

(72) Daponte, Diego H., US

(72) Shawver, Susan E., US

(72) Watkins, Sharon L., US

(72) Watts, Hugo P., GB

(73) Kimberly-Clark Corporation, GB

(51) Int. Cl.<sup>6</sup> D04H 3/03, B32B 5/02, D04H 3/16

(30) 1990/05/04 (518,904) US

(30) 1990/12/13 (626,817) US

(54) **TUILES EN POLYMERE, A TROIS DIMENSIONS,  
PRESENTANT DES CARACTERISTIQUES PHYSIQUES  
AMELIOREES**

(54) **NONWOVEN WEBS OF DRAWN AND UNORIENTED  
THERMOPLASTIC FIBERS**

(57) On présente un voile non tissé tridimensionnel constitué de fibres thermoplastiques étirées et non orientées formé à partir d'un mélange de polypropylène et de polybutylène, où le mélange est constitué, en poids, de 90 % à 70 % de polypropylène et de 10 % à 30 % de polybutylène. Le mélange peut être un mélange d'un homopolymère de polypropylène et d'un homopolymère de polybutylène; d'un homopolymère de polypropylène et d'un copolymère de polybutylène; d'un copolymère de polypropylène et d'un homopolymère de polybutylène; et d'un copolymère de polypropylène et d'un copolymère de polybutylène. De plus, des mélanges ternaires comprenant du polypropylène, du polybutylène et un terpolymère (propylène, éthylène, et 1-butène) sont utiles. Les voiles non tissés ont une résistance, une ténacité et une résistance à la déchirure accrues.

(57) There is disclosed a three dimensional nonwoven web consisting of drawn and unoriented thermoplastic fibers formed from a blend of polypropylene and polybutylene, wherein the blend by weight is from 90% to 70% polypropylene and from 10% to 30% polybutylene. The blend can be a blend of a homopolymer of polypropylene and a homopolymer of polybutylene; a homopolymer of polypropylene and a copolymer of polybutylene; a copolymer of polypropylene and a homopolymer of polybutylene; and a copolymer of polypropylene and a copolymer of polybutylene. In addition, ternary blends comprising polypropylene, polybutylene, and a terpolymer (propylene, ethylene, and 1-butene) are useful. The resulting nonwoven webs have enhanced strength, toughness, and tear resistance.



## **THREE-DIMENSIONAL POLYMER WEBS WITH IMPROVED PHYSICAL PROPERTIES**

### **Background of the Invention**

This invention relates generally to three dimensional nonwoven webs consisting of thermoplastic fibers, and more particularly concerns such webs which are formed from blends of polypropylene and polybutylene.

Nonwoven webs formed of thermoplastic fibers are well known in the art and have found uses in a variety of applications. In one application, such nonwoven webs are formed by melt-blowing and are used as disposable industrial wipers. Such industrial wipers find application in numerous manufacture and maintenance facilities where personnel find it necessary to wipe up oil, grease, and water from a variety of surfaces. One such wiper made of melt-blown polypropylene fibers is manufactured and sold by Kimberly-Clark Corporation, the assignee of the present invention, under the trademark Kintex®.

Melt-blown nonwoven wipers of polypropylene thermoplastics fibers have advantage over cloth wipers in being cost effectively disposable with similar wiping characteristics as compared to cloth. Particularly, industrial wipers must be able to quickly pick up spilled liquids, both oil based and water based, and leave a clean, streak free

surface. In addition, the wipers must have sufficient capacity to hold such liquids within the wiper structure until it is desired to remove the liquid by pressure such as by wringing.

5 Nonwoven melt-blown industrial wipers formed from polypropylene in the past have performed adequately in terms of their wiping characteristics, particularly with respect to oil and, when treated with a surfactant, with respect to water. Moreover, nonwoven industrial wipers  
10 made of polypropylene fibers have exhibited resistance to most commercially available solvents.

A superior three dimensional nonwoven melt-blown wiper, however, could be achieved by increasing strength, increasing toughness, and increasing tear resistance. A  
15 three dimensional nonwoven web is described in U.S. Patent No. 4,741,941, to Englebert. However, the web described in Englebert does not teach the increased strength which characterizes a superior three dimensional nonwoven melt-blown wiper.

20 Basically, the present invention relates to a web including at least a layer consisting of drawn and unoriented thermoplastic fibers formed from a blend by weight of from 90% to 70% polypropylene polymers and from 10% to 30% butylene polymers.

25 The polymers may be selected from the group consisting of homopolymers and copolymers of propylene and polymers selected from the group consisting of copolymers of butylene; or copolymers of propylene and polymers selected from the group consisting of homopolymers and  
30 copolymers of butylene.

The invention may take the form of a three dimensional nonwoven web, or a nonwoven wiper, including at least one such layer. It may also be in the form of a

laminate material comprising at least one such nonwoven web, and there may be provided a three dimensional nonwoven wiper comprising a laminate material including one such layer in a three dimensional form.

5 Another aspect of the invention resides in a method of forming a nonwoven material including the steps of heating a blend of the type described above to form a melt and extruding the melt through a die to form thermoplastic fibers. The fibers are initially drawn to a ratio of  
10 greater than 14 to 1, and they are deposited onto a forming surface to form a first layer.

The forming surface may be shaped to produce a three dimensional material, and there may be included the additional step of bonding the material to the second or  
15 third layer to form a laminate. A three dimensional nonwoven web of the described type will display improved strength, toughness, and tear resistance over three dimensional melt-blown webs formed of polypropylene and polyethylene fibers.

20 An industrial wiper comprising the three dimensional nonwoven melt-blown web consisting of thermoplastic fibers provides improved strength, toughness, and tear resistance over three dimensional melt-blown wipers formed of polypropylene and polyethylene fibers.

25 A laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers provides improved strength, toughness, and tear resistance over laminate materials which contain at least one three dimensional layer formed of polypropylene and polyethylene  
30 fibers.

An industrial wiper formed from a laminate material comprising at least one three dimensional layer consisting of thermoplastic fibers provides improved strength, toughness, and tear resistance over industrial wipers  
35 formed from laminate materials which contain at least one

three dimensional layer formed of polypropylene and polyethylene fibers.

In one form of the invention, the nonwoven webs of the present invention consists of drawn and unoriented thermoplastic fibers formed from blends of polypropylene and polybutylene including blends of homopolymers of polypropylene and homopolymers of polybutylene; from blends of homopolymers of polypropylene and copolymers of polybutylene; from blends of copolymers of polypropylene and homopolymers of polybutylene; and from blends of copolymers of polypropylene and copolymers of polybutylene.

In addition, the objects of the present invention can be obtained by nonwoven webs consisting of drawn and unoriented thermoplastic fibers formed from a ternary blend comprising polypropylene, polybutylene and a terpolymer comprising propylene, ethylene, and 1-butene.

In connection with the present invention, "drawn thermoplastic fibers" refers to fibers that are drawn to a ratio of generally greater than 14 to 1 and optionally greater than 50 to 1 in the forming process. Also in connection with the present invention, "unoriented thermoplastic fibers" refers to fibers which solidify in a relaxed condition (not under tension). Such drawn and unoriented fibers are characteristically formed by melt-blowing techniques as well as other fiber forming techniques such as melt-spraying.

More particularly, the objectives of the present invention are realized by a three dimensional nonwoven melt-blown web consisting of drawn and unoriented thermoplastic fibers formed by melt-blowing a blend comprising polypropylene and polybutylene. Particularly, the three dimensional blend of polypropylene and polybutylene is from 90-70% by weight of polypropylene and from 10-30% by weight of polybutylene. More particularly, in the case of a nonwoven wiper, the three dimensional

blend is preferred to be from 85% to 75% by weight of polypropylene and from 15% to 25% by weight of polybutylene.

5 In connection with the present invention, the prior art European Patent Application No. 89303407.4 of Don & Low Ltd. has disclosed the usefulness of blending polypropylene and polybutylene to form fibers, tapes, and films where the thermoplastic fibers, tapes, and films are drawn to a ratio of at least 8:1 to produce an oriented  
10 molecular structure. Particularly, the Don & Low reference discloses thermoplastic fibers, tapes and films that have improved strength as measured by tenacity which is the maximum stress they can resist without rupture. Such thermoplastic fibers, tapes, and films result from  
15 blending up to 10% by weight of polybutylene with polypropylene with from 2% to 4% by weight of polybutylene being preferred. The Don & Low reference, however, teaches that "it has been found that if the polybutylene proportion is increased beyond ten percent there is little  
20 if any increase in strength [tenacity]". Also the Don & Low reference does not teach improved elongation. As will be demonstrated hereinbelow the nonwoven melt-blown and melt-spray webs of the present invention result from a blend of polypropylene and polybutylene with the  
25 polybutylene in excess of 10%. Moreover, the thermoplastic fibers of the

present invention are drawn to a ratio of substantially greater than 14 to 1 and are not oriented as a result of the melt-blowing or melt-spraying process.

5        **Brief Description of the Drawings**

Fig. 1 is a schematic diagram showing showing machinery for producing a three dimensional nonwoven melt-blown web in accordance with the present invention.

10       **Detailed Description of the Invention**

While the invention will be described in connection with a preferred embodiment and procedure, it will be understood that we do not intend to limit the invention to that embodiment or procedure. On the contrary, we intend to cover all alternatives, modifications, and equivalents as may be included within the spirit and scope of the invention as defined by the appended claims.

Turning to Fig. 1 there is shown a web forming machine 10 for forming a melt-blown web 12 made up of a number of layers of melt-blown fibers 50. The machine 10 includes eight identical extruders 14A-H with corresponding hoppers 16A-H for receiving thermoplastic resin pellets. The extruders 14A-H include internal screw conveyors which are driven by motors 15A-H. The extruders 14A-H are heated along their lengths to the melting temperature of the thermoplastic resin pellets to form a melt. The screw conveyors driven by motors 15A-H force the thermoplastic material through the extruders into attached delivery pipes 20A-H which are connected to die heads 22A-H, each having a die width 25.

Die head 22A, for example, comprises a die tip 24 which has a die opening or orifice (not shown). The die tip may be recessed, flush, or stick out. Hot fluid, usually air, is supplied to the die tip via pipes 32 and 34 (Fig. 1) which terminate in channels adjacent to the orifice of the die tip.

As the thermoplastic polymer exits the die tip for each die head, the high pressure air attenuates and breaks up the polymer stream to form fibers at each die head. The fibers are then in accordance with the present invention quenched with a mist of water from spray nozzles 27A-H. The spray nozzles are located just below the orifice of the die tip and spray the fibers 50 with water at room temperature or slightly above at a flow rate of at least 0.02 liter/min/inch width of die tip. Fiber quenching is conventional as demonstrated by United States Patent No. 3,959,421. If desired, a surfactant can be added to the fibers by adding the surfactant to the quenching water. It should also be noted that significantly improved toughness and tear resistance result only if the fibers are subjected to quenching.

Once the fibers have been quenched, they are deposited on a forming surface 38 to form the layered web. In the preferred embodiment of the present invention, the forming surface 38 is an open mesh wire surface into which the fibers are pulled in order to form the three dimensional layer. However, a number of alternative types of forming surfaces 38 may be utilized in order to produce the desired three dimensional layer. A variety of such forming surfaces are described in the Englebert patent and are well-known in the art.

A vacuum is drawn behind the forming surface 38 to draw the fibers onto the forming surface 38 during the process of melt-blowing. Separate vacuum chambers behind the forming surface 38 may be provided for each die head 22A-H. Once the fiber layers have been deposited on the forming surface 38 by the multiple die heads 22A-H, the web 12 is drawn from the forming surface 38 by withdrawal rolls 40 and 42. Embossing rolls 44 and 46 engage the web 12 after the withdrawal rolls to emboss the web with a pattern.



5 The foregoing description of the melt-blowing machine 10 is generally conventional and well known in the art as demonstrated by NRL Report 4364, "Manufacture of Super-Fine Organic Fibers", by V.A. Wendt, E.L. Boon, and C.D. Fluharty; NRL Report 5265, "An Improved Device for the Formation of Super-Fine Thermoplastic Fibers", by K.D. Lawrence, R.T. Lukas, and J.A. Young; and United States Patent 3,849,241, issued November 19, 1974, to Buntin, et al. It also will be appreciated by one of ordinary skill in the art that a single head melt-blowing machine can be used instead of the multiple bank machine illustrated. It should also be appreciated that the web 12 may be comprised of a single layer, multiple layers which are all identical in composition, or multiple layers some of which layers are made in accordance with the present invention and some of which layers are conventional. Moreover, ordinary skill in the art will appreciate that fine adjustment of the equipment and process may be required to optimize performance and efficiency. Such fine adjustment can be accomplished by one of ordinary skill without undue experimentation.

25 In addition, the fibers and the resulting web can be formed by other fiber forming techniques including, for example, melt-spray techniques. Melt-spraying is a process for forming fibers from a fiber forming resin using compact spray head designs usually including one to four spray heads in a cluster. The equipment includes a die housing with a hydraulic chamber and a retractable piston assembly for releasing molten resin. As the molten resin is released, it is contacted and drawn by a primary air supply which completely surrounds the molten resin and contacts it at a predetermined angle. If additional drawing and attenuation of the newly formed fibers is desired, secondary fiberization air also may be utilized. The secondary fiberization air will most typically include at least two fluid

streams which each impinge upon the resin/fibers at a second angle.

5 In more refined embodiments the fiberization air can also be angled such that it will spiral around the forming fibers. Additionally, the piston within the hydraulic chamber may be cycled on and off to interrupt the flow of the fiber forming resin thereby creating discrete pluralities of fibers.

10 Both melt-blowing and melt-spraying techniques produce fibers that are drawn and unoriented.

In accordance with the present invention, it has been found that an improved three dimensional nonwoven web of drawn and unoriented thermoplastic fibers can be formed by melt-blowing a blend of polypropylene and polybutylene to enhance certain properties in the melt-blown web 12, as compared with 100% three dimensional melt-blown polypropylene and polyethylene. Specifically, the melt-blown web 12 formed from melt-blowing a blend of polypropylene and polybutylene improves the strength, toughness (the elongation and absorbed energy), and tear resistance. Particularly, we have found that blends of from 90% to 70% polypropylene and from 10% to 30% polybutylene produce three dimensional nonwoven melt-blown webs with these improved characteristics. The advantages of the present invention can be realized by blending homopolymers of polypropylene and polybutylene; by blending copolymers of polypropylene and homopolymers of polybutylene; by blending homopolymers of polypropylene and copolymers of polybutylene; and by blending copolymers of polypropylene and copolymers of polybutylene. The copolymers of polypropylene may have ethylene in amounts ranging up to about 3% by weight. The copolymers of polybutylene may have ethylene in amounts ranging up to about 6% by weight.

35

In addition, the invention may be carried out by using ternary blends comprising polypropylene, polybutylene, and a terpolymer (propylene, ethylene, and 1-butene). The resulting three dimensional nonwoven webs have enhanced strength, toughness, and tear resistance.

Webs in accordance with the present invention can be formed at polymer throughputs of from 1 to 15 pounds per inch of die head width per hour (pih). Once the melt-blown web has been formed, the material may be bonded or unbonded. The bond pattern may be a line pattern, a weave pattern, or a point pattern, but the point pattern, with bonding occurring within certain discrete areas on the material, is preferred. The bonding may be accomplished by ultrasonic heating, by use of an adhesive, or by embossing with heated rolls. In connection with the present invention, most webs described in the following examples were heat embossed with a weave pattern, such as that shown in United States Patent Des. 264,512, or with a dot pattern. The resulting bonded webs had a bonded area of about 18% of the total area when embossed with the weave pattern and of 5% - 18% when embossed with the dot pattern.

In carrying out the present invention, the blends and ternary blends were formed by simply mixing the thermoplastic pellets in the proper weight proportions before being added to the hoppers 16A-H. The terpolymers were polymerized in the desired proportions during manufacture.

Three dimensional nonwoven webs formed from blends, ternary blends, terpolymers, and terpolymer blends utilizing the present invention were made and tested in accordance with the following examples which illustrate the invention.

### Example 1

5 A melt-blowing machine similar to that shown in Figure 1 was used to form a control web, Sample 59, from 100% polypropylene and four additional webs, Samples 61, 62, 64, and 66, from blends of 90% polypropylene and 10% polybutylene, 85% polypropylene and 15% polybutylene, 80% polypropylene and 20% polybutylene, and 70% polypropylene and 30% polybutylene, respectively. The polypropylene used for the control web, Sample 59, was a homopolymer with a melt flow of 800g/10min (ASTM D1288, Condition L, (230°C, 2160g weight)) and a narrow molecular weight distribution. Such a polypropylene homopolymer is manufactured by Himont U.S.A., Inc of Wilmington, Delaware and designated Valtec HH442H. The polypropylene used for the inventive melt-webs, Samples 61, 62, 64, and 66, was also Valtec HH442H. The polybutylene used for Samples 61, 62, 64, and 66 was Duraflex DP-8910, which is an ethylene copolymer (6% ethylene) and is manufactured by Shell Chemical Company of Houston, Texas. The melt-blowing machine was set up in accordance with the following process conditions for Samples 59, 61, 62, 64, and 66:

#### Machine Configuration:

- A. recess die tip
- B. single bank
- C. water quench with surfactant

#### Set Points:

		<u>Sample</u> <u>59</u>	<u>Samples</u> <u>61- 66</u>
35 Extruder Barrel Pressure	psi	500	500
Primary Air Pressure	psi	2.4	2.2
Primary Air Temperature	°F	583	565
Forming Distance	in	6.5	6.0

Measured Variables:

Die Tip Pressure	psi	21	15
Die Tip (Melt) Temperature	°F	497	489

5                    In order to provide an accurate comparison between  
conventional Sample 59 and inventive Samples 61, 62, 64,  
and 66, the actual data for the conventional web, Sample 59,  
and the inventive webs, Samples 61, 62, 64, and 66, has  
10                   been normalized to a basis weight of 1 gsm. Both the actual  
data and the normalized data which show the characteristics  
of Samples 59, 61, 62, 64, and 66 are set out in Table 1  
below.

TABLE I -- COMPARISON WITH 100% POLYPROPYLENE  
POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELT-BLOWN

Sample Number	Composition	GRAB TENSILE						TRAP TEAR			NORMALIZED GRAB TENSILE					
		Basis Weight gsm	Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	Load MD g	Load CD g	Load MD g/gsm	Load CD g/gsm	Load MD g/gsm	Load CD g/gsm	Load MD g/gsm	Load CD g/gsm
59	100% PP	42	1344	1453	26	46	30	50	245	227	32	35	6	5		
61	90:10 PP:PB	25	1544	1444	73	62	75	72	454	368	63	59	18	15		
62	85:15 PP:PB	41	1839	1562	87	70	76	73	558	327	45	38	14	8		
64	80:20 PP:PB	25	1880	1775	53	60	46	55	545	381	76	72	22	15		
66	70:30 PP:PB	26	2393	1525	52	41	30	42	849	420	91	58	32	16		

In connection with the characteristics reported in Table 1, the basis weight was measured in accordance with Federal Test Method 191A-5 and expressed in grams per square meter (gsm). The bulk was measured in accordance with the Ames Method and expressed in millimeters (mm). The grab tensile strength was measured in accordance with Method 5100, Federal Test Method 191A and expressed in grams (g), kilogram-millimeters (kg-mm), and millimeters (mm). The trapezoid tear was measured in accordance with Method 5135, Federal Test Method 191 and expressed in grams (g).

In order to measure strength and toughness, the various samples were subjected to tensile testing in the machine direction and the cross-machine direction. Toughness is determined by the amount of energy that the material will absorb prior to failure. The peak energy is the amount of energy the material will absorb until the peak load is achieved. The fail energy is the amount of total energy the material will absorb until it finally fails by separating. Particularly as the stress to which the web is subjected is increased, the web begins stretching or elongating. At some point, the web reaches a peak loading at which failure begins and additional stressing does not increase the loading on the web. As the material begins failing, further elongation results under decreasing loading. The amount of peak energy the material experiences is the integral of the load v. elongation curve for the web from 0 load to the peak of the load. The amount of failed energy is the integral of the load v. elongation curve for the web from the initial 0 load until rupture occurs and the load again returns to 0. Both peak energy and fail energy give a good indication of the toughness of a web. Peak strength is determined by measuring the maximum load achieved before the web begins to fail.

Consequently, it can be seen from Table 1 that Samples 61 - 66, made in accordance with the present invention, all demonstrate a substantial increase in the peak load and energy over those of the prior art 100% three dimensional polypropylene melt-blown web of Sample 59. Particularly, Samples 61 - 66 have an increase in peak load in the cross-machine direction of between 15% and 78% over the control Sample 59. Additionally, Samples 61 - 66 have an increase in peak energy in the machine direction of between 100% and 215% over the control Sample 59. Accordingly, the inventive webs, Samples 61 - 66, all demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

While toughness measures a web's resistance to initial tearing, trapezoid tear measures the web's resistance to the propagation of a tear after an initial tear. As can be seen again from Table 1, Samples 61 - 66 show between a 85% and 247% increase in their tear resistance over that of the control Sample 59.

### Example 2

Inventive webs, Samples 61 - 66, were next compared to a control web, Sample 2, formed from 100% polyethylene. The polyethylene used for the control web, Sample 2, was a copolymer (with 1-octene) with a melt index of 135 (ASTM D1288, Condition E, (190°C, 2160g weight)) and a narrow molecular weight distribution. Such a polyethylene homopolymer is manufactured by Dow Chemical, Inc. of Wilmington, Delaware and designated ASPUN® 6814A. The melt-blowing machine was set up in accordance with the following process conditions for Sample 2:



Machine Configuration:

- A. recess die tip
- B. single bank
- C. water quench with surfactant

5

Set Points:

Extruder Barrel Pressure	psi	504
Primary Air Pressure	psi	4.2
Primary Air Temperature	°F	507
Forming Distance	in	7.5

10

Measured Variables:

Die Tip Pressure	psi	139
Die Tip (Melt) Temperature	°F	507

15

As with Example 1, the actual data for the conventional web, Sample 2, and the inventive webs, Samples 61, 62, 64, and 66, has been normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of Samples 2, 61, 62, 64, and 66 are set out in Table 2 below.

20

TABLE 2 -- COMPARISON WITH 100% POLYETHYLENE  
POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	GRAB TENSILE						TRAP TEAR			NORMALIZED GRAB TENSILE				NORMALIZED TRAP TEAR			
		Basis Weight gsm	Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	Load MD g	Load CD g	Load MD gsm	Load CD gsm	Load MD gsm	Load CD gsm	Load MD gsm	Load CD gsm	Load MD gsm	Load CD gsm
2	PE	40	985	908	44	55	67	97			25	23						
61	90:10 PP:PB	25	1544	1444	73	62	75	72	454	368	63	59	18				15	
62	85:15 PP:PB	41	1839	1562	87	70	76	73	558	327	45	38	14				8	
64	80:20 PP:PB	25	1880	1775	53	60	46	55	545	381	76	72	22				15	
66	70:30 PP:PB	26	2393	1525	52	41	30	42	849	420	91	58	32				16	

5 It can be seen from Table 2 that Samples 61 - 66,  
made in accordance with the present invention, all  
demonstrate a substantial increase in the peak load and  
energy over those of the prior art 100% three dimensional  
polyethylene melt-blown web of Sample 2. Particularly,  
Samples 61 - 66 have an increase in peak load in the  
machine direction of between 57% and 143% over the  
control Sample 2. Additionally, Samples 61 - 66 have an  
increase in peak energy in the machine direction of between  
10 18% and 98% over the control Sample 2. Accordingly, the  
inventive webs. Samples 61 - 66, all demonstrate the  
increased strength and toughness of the three dimensional  
nonwoven webs prepared in accordance with the present  
invention.

### 15 Example 3

In order to determine the effectiveness of the ternary  
blends of the present invention, Samples 4, 6, 7 and 8 were  
20 prepared in accordance with the present invention for  
comparison with control Samples 2 and 59. Samples 4 and  
6 were formed from a blend of 50% polypropylene  
(homopolymer; Himont HH442H) and 50% terpolymer  
(formed from three monomers, namely propylene,  
25 ethylene, and 1-butene), which was an experimental  
polymer manufactured by Himont U.S.A., Inc. of  
Wilmington, Delaware, identified by the designation 9582-  
35-1, having a melt flow rate of 45 g/10 min. Samples 7  
and 8 are examples of a ternary blend of polymers in  
30 accordance with the present invention. Particularly,  
Samples 7 and 8 were formed from a blend of 45%  
polypropylene (homopolymer; Himont HH442H), 45%  
terpolymer (same terpolymer as present in Samples 4 and  
6), and 10% polybutylene (Shell DP8910).

Samples 4, 6, 7, and 8 were formed by melt-blowing in accordance with the following parameters:

Machine Configuration:

- 5      A. recess die tip  
        B. single bank  
        C. water quench with surfactant

Set Points:

		<u>Samples</u> <u>4/6</u>	<u>Samples</u> <u>7/8</u>
10	Primary Air Pressure	psi 3	2.5
	Primary Air Temperature	°F 623	635
15	Forming Distance	in 7	7

Measured Variables:

	Die Tip Pressure	psi 125	128
20	Die Tip (Melt) Temperature	°F 579	557

25      As with the previous examples, the actual data for the inventive webs, Samples 4, 6, 7, and 8, has been normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of Samples 2, 4, 6, 7, 8, and 59 are set out in Table 3 below.

TABLE 3 -- COMPARISON WITH TRIBLEND PP:PB:TERPOLYMER  
POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS  
IN THREE DIMENSIONAL MELTBLOWN

Sample Number	Composition	GRAB TENSILE						NORMALIZED GRAB TENSILE		
		Basis Weight gsm	Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	Load MD g/gsm	Load CD g/gsm
7 & 8	45:45:10 PP:TERP:PB	41	1871	1738	85	98	63	85	46	42
4 & 6	50:50 PP:TERP	41	2395	1841	53	67	28	51	59	45
59	100% PP	42	1344	1453	26	46	30	50	32	35
2	100% PE	40	985	908	44	55	67	97	25	23

It can be seen from Table 3 that although Samples 4 and 6, made in accordance with the present invention, demonstrate a substantial increase in the peak load and energy over those of the prior art three dimensional melt-blown webs of Samples 2 and 59, the polypropylene/terpolymer blend has a decreased elongation, which is an important characteristic of toughness. To the contrary, Samples 7 and 8 demonstrate increased strength, toughness, and elongation. Particularly, Samples 7 and 8 have an increase in peak load in the machine direction of between 39% and 90% over the control Samples 2 and 59. Additionally, Samples 7 and 8 have an increase in peak energy in the machine direction of between 93% and 227% over the control Samples 2 and 59, an increase in elongation of between 70% and 110% compared with control Sample 59, and a comparable elongation with that of control Sample 2. Accordingly, the inventive ternary blend webs, Samples 7 and 8, demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

#### Example 4

In order to test the effectiveness of the three dimensional web of the present invention as a laminate material, Samples 53, 56, 213, C, D were prepared in accordance with the present invention. Each of these samples was bonded to an intermediate layer of material which was not the three dimensional web of the present invention.

Samples 55 and 207 were also prepared to show the effectiveness of the three dimensional web of the present invention as a laminate material in situations where the three dimensional web will be directly bonded to another three dimensional web, without any intermediate layer.

5        Sample 53 was formed from a blend of 60%  
polyethylene (Dow 6814) and 40% polypropylene. The  
polypropylene used for Sample 53 was a homopolymer with  
a melt flow of 400g/10min (ASTM D1288, Condition L,  
(230°C, 2160g weight)) and a narrow molecular weight  
distribution. Such a polypropylene homopolymer is  
manufactured by Himont U.S.A., Inc. of Wilmington,  
Delaware and designated HH441. Sample 56 was formed  
from a blend of 75% polyethylene (Dow 6814) and 25%  
10       polypropylene (Himont HH441). As an intermediate  
material to which the laminate materials of Samples 53 and  
56 were bonded, Intermediate Layer X was formed from a  
blend of 60% pulp (IPSS) and 40% ethylene vinyl acetate  
copolymer (Exxon "Escorene").

15       Sample 213 was formed from a blend of 25%  
polybutylene (Shell DP8910) and 75% polypropylene  
(Himont HH442H) and was bonded to an intermediate  
material "X," formed from a blend of 70% pulp (IPSS) and  
30% ethylene vinyl acetate copolymer (Exxon "Escorene").

20       Sample C was formed from a blend of 80%  
polypropylene (Himont HH442H) and 20% polybutylene  
(Shell DP8910) which was bonded to an intermediate  
material "X," formed from a blend of 30% pulp  
(Weyerhaeuser NF105) and 70% polymer blend (comprised  
25       of 85% polypropylene (Himont HH442H) and 15%  
polybutylene (Shell DP8910)).

30       Sample D was formed from a blend of 80%  
polypropylene (Himont HH442H) and 20% polybutylene  
(Shell DP8910) which was bonded to an intermediate  
material "X," formed from a blend of 50% pulp  
(Weyerhaeuser NF105) and 50% polymer blend (comprised  
of 85% polypropylene (Himont HH442H) and 15%  
polybutylene (Shell DP8910)).

35       Sample 55 was formed from a blend of 75%  
polyethylene (Dow 6814) and 25% polypropylene (Himont

HH442H). Sample 207 was formed from a blend of 80% polypropylene (Himont HH442H) and 20% polybutylene (Shell DP8910).

All the webs were heat embossed with a conventional bonding pattern.

Samples 53, 56, 213, C, D, 55, and 207 were formed by melt-blowing in accordance with the following parameters:

Machine Configuration:

- A. recess die tip
- B. single bank
- C. water quench with surfactant

		3D Layer <u>Sample 53</u>	Intermediate Layer <u>X</u>
	Primary Air Pressure	psi 1.5	4.6
	Primary Air Temperature	°F 512	404
	Forming Distance	in 6.5	9
	Primary Air Flow	scfm 168	
	<u>Measured Variables:</u>		
	Die Tip Pressure	psi 132	
	Die Tip (Melt) Temperature	°F 589	381
	<u>Bonding:</u>		
	Pattern Roll Temperature		
	Set Point	°F 180	
	Actual Surface	°F 160	
	Anvil Roll Temperature		
	Set Point	°F 180	
	Actual Surface	°F 170	
	Pressure (Average)	psi 10	
	Line Speed	fpm 14	
		3D Layer <u>Sample 56</u>	Intermediate Layer <u>X</u>
	Primary Air Pressure	psi 4.6	4.6
	Primary Air Temperature	°F 545	404
	Forming Distance	in 9	9
	Primary Air Flow	scfm 400	



Measured Variables:

Die Tip Pressure	psi	174	
Die Tip (Melt) Temperature	°F	538	381

5

Bonding:

Pattern Roll Temperature		
Set Point	°F	180
Actual Surface	°F	160

10

Anvil Roll Temperature		
Set Point	°F	180
Actual Surface	°F	170

15

Pressure (Average)	psi	10
Line Speed	fpm	14

20

3D Layer  
Sample 213Intermediate Layer  
X

Primary Air Pressure	psi	2.3	5.0
Primary Air Temperature	°F	511	380
Forming Distance	in	7.5	
Primary Air Flow	scfm	66	

25

Measured Variables:

Die Tip Pressure	psi		220
Die Tip (Melt) Temperature	°F	536	370
(and Triton X-102 surfactant quench)			

30

Bonding:

Pattern Roll Temperature		
Set Point	°F	185
Actual Surface	°F	160

35

Anvil Roll Temperature		
Set Point	°F	190
Actual Surface	°F	173

40

Pressure (Average)	psi	12.5
Line Speed	fpm	21

45

3D Layer  
Sample CIntermediate Layer  
X

Primary Air Pressure	psi	1.6	6.3
Primary Air Temperature	°F	517	514
Forming Distance	in	7	18
Primary Air Flow	scfm		

50

Measured Variables:

5	Die Tip Pressure	psi	79	117
	Die Tip (Melt) Temperature	°F	495	514
	(and Triton X-102 surfactant quench)			

Bonding:

10	Pattern Roll Temperature			
	Set Point	°F	220	
	Actual Surface	°F		

15	Anvil Roll Temperature			
	Set Point	°F	230	
	Actual Surface	°F		

15	Pressure (Average)	psi	16	
	Line Speed	fpm	22	

20		3D Layer	Intermediate Layer	
		D	X	
25	Primary Air Pressure	psi	1.6	6.3
	Primary Air Temperature	°F	517	514
	Forming Distance	in	7	18
	Primary Air Flow	scfm		

Measured Variables:

30	Die Tip Pressure	psi	79	117
	Die Tip (Melt) Temperature	°F	495	514
	(and Triton X-102 surfactant quench)			

Bonding:

35	Pattern Roll Temperature			
	Set Point	°F	220	
	Actual Surface	°F		

40	Anvil Roll Temperature			
	Set Point	°F	230	
	Actual Surface	°F		

40	Pressure (Average)	psi	16	
	Line Speed	fpm	22	

25

		3D Layer <u>55</u>	3D Layer <u>207</u>
--	--	-----------------------	------------------------

5	Primary Air Pressure	psi	1.5
	Primary Air Temperature	°F	545
	Forming Distance	in	7
	Primary Air Flow	scfm	400

Measured Variables:

10	Die Tip Pressure	psi	174	56
	Die Tip (Melt) Temperature	°F	538	502
	(and Triton X-102 surfactant quench)			

Bonding:

15	Pattern Roll Temperature			
	Set Point	°F	180	237
	Actual Surface	°F	160	207
	Anvil Roll Temperature			
20	Set Point	°F	180	224
	Actual Surface	°F	170	202
	Pressure (Average)	psi	10	22
	Line Speed	fpm	14	39

25

As with the previous examples, the data for these inventive samples was all normalized to a basis weight of 1 gsm. Both the actual data and the normalized data which show the characteristics of these inventive samples are set out in Table 4 below.

30

TABLE 4 -- COMPARISON OF COMPOSITES WITH 3D OUTER LAYERS

## POLYMER BLENDS FOR IMPROVED STRENGTHS AND TOUGHNESS IN THREE DIMENSIONAL MELTLOWN

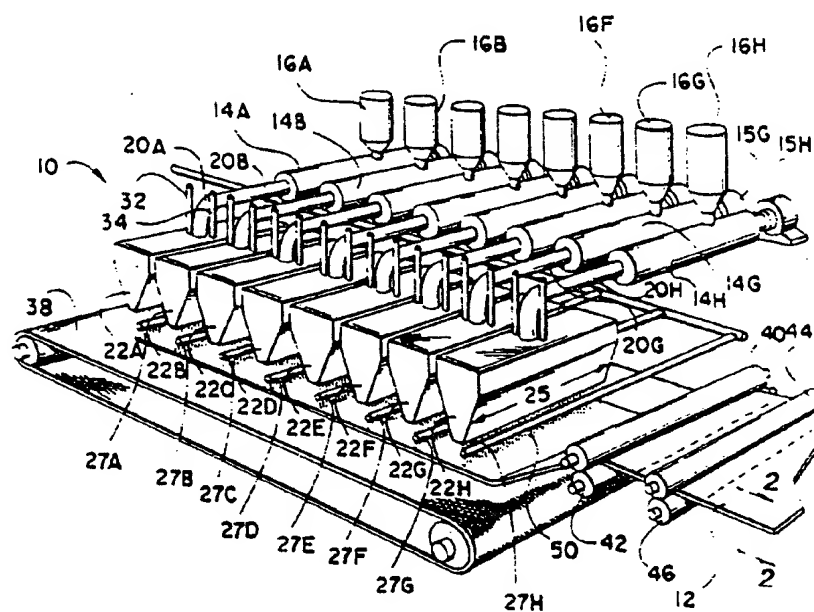
Sample Number	Composition	GRAB TENSILE								TRAP TEAR				NORMALIZED GRAB TENSILE								NORMALIZED TRAP TEAR			
		Basis Weight, gsm	Load MD g	Load CD g	Energy MD kg-mm	Energy CD kg-mm	Elong MD mm	Elong CD mm	Load MD g	Load CD g	Elong MD mm	Elong CD mm	Load MD g/gsm	Load CD g/gsm	Load MD g/gsm	Load CD g/gsm	Load MD g/gsm	Load CD g/gsm	Load MD g/gsm	Load CD g/gsm					
<u>D + x + 3D</u>																									
53	3D = 60:40 PE:PP x = 60:40 PULP:EVA	80	1895	1960	85	98	69	80	530	617	24	25	7	8											
56	3D = 75:25 PE:PP x = 60:40 PULP:EVA	103	1651	1878	39	77	26	40	522	270	16	18	5	3											
<u>213</u>																									
	3D = 75:25 PP:PB x = 70:30 PULP:EVA	88	2301	2117	145	161	85	97	828	816	26	24	9	9											
<u>C</u>																									
	3D = 80:20 PP:PB 3D x = 30:70 PULP: (85:15 PP:PB)	111	5269	4272	175	182	44	63	1221	653	47	38	11	6											
<u>D</u>																									
	3D = 80:20 PP:PB 3D x = 30:70 PULP: (85:15 PP:PB)	112	4217	3314	117	108	38	46	980	648	38	30	9	6											
<u>D + 3D (no intermediate layer)</u>																									
55	3D = 75:25 PE:PP	76	2118	1689	63	32	31	29	573	226	28	22	8	3											
207	3D = 80:20 PP:PB	50	2334	1856	111	108	71	89	892	661	47	37	18	13											

It can be seen from Table 4 that the laminate material formed from polyethylene, namely samples 53 and 56, do not exhibit the same strength, toughness, and tear resistance as those formed from polybutylene, namely Samples 213, C, and D. In particular, the polybutylene laminate materials bonded to an intermediate layer have an increase in peak load in the machine direction of 121% on average over the polyethylene laminate materials. Additionally, these polybutylene laminate materials have an increase in peak energy in the machine direction of 135% on average over the polyethylene laminate materials. Accordingly, the inventive laminate materials containing polybutylene, Samples 213, C, and D demonstrate the increased strength and toughness of the three dimensional nonwoven webs prepared in accordance with the present invention.

As can also be seen from Table 4, Samples 213, C, and D formed with polybutylene, show much greater tear resistance than the samples formed with polyethylene.

It can also be seen from Table 4 that the three dimensional layer formed with polybutylene, Sample 207, had a 10% greater peak load than the three dimensional layer formed with polyethylene, Sample 55. As for peak energy, Sample 207 was 76% greater than Sample 55. Additionally, Sample 207's tear resistance was 56% greater than the tear resistance of Sample 55. It should be noted that there was no intermediate layer bonded between the two three dimensional layers of Samples 207 and 55.

2038754



**Fig. 1**

Scott & Aylen